there is considerable dispersion in ω *vs.* q owing to intermolecular coupling in the crystal lattice, infrared measurements on solids are not necessarily expected to agree closely with neutron results.

IV. Conclusion

The H_2O librational and translational bands assigned from the present neutron study on a series of transition metal hydrates are generally consistent with previous infrared and structural results. The average strength of binding of the water molecules (as indicated by the H_2O bands) does not appear to change significantly in proceeding from the higher to the lower hydrates. A comparison between the librational bands in $UO_2(NO_3)_2 \cdot 6H_2O$ and the other higher hydrates indicates somewhat weaker binding for the H_2O molecules in the uranyl salt. It is probable that neutron-scattering measurements with higher resolution would result in better separation of the broad, overlapping peaks in the neutron spectra.

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The Reactions of N,N-Bis(trifluoromethyl)hydroxylamine with Perfluoroacyl and Carbonyl Halides

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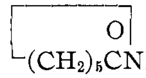
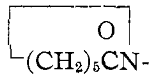
N,N-Bis(trifluoromethyl)hydroxylamine reacts with either CsF or KF to form a solid adduct. This adduct reacts with COF_2 to form $[(CF_3)_2NO]_2CO$ and $(CF_3)_2NOC(O)F$ and with $COCl_2$ to form $(CF_3)_2NOC(O)Cl$. $(CF_3)_2NOC(O)CF_3$ and $(CF_3)_2NOC(O)C_3F_7$ are obtained with the perfluoroacyl chlorides, $CF_3C(O)Cl$ and $C_3F_7C(O)Cl$, respectively. These compounds have been characterized and the N,N-bis(trifluoromethyl)hydroxylamine–metal halide adduct is discussed.

Very few of the reactions of N,N-bis(trifluoromethyl)hydroxylamine,[1] $(CF_3)_2NOH$, have been investigated. It reacts with a variety of substances to give the bis(trifluoromethyl)nitroxide radical[2-4] and with PCl_5 to give bis(trifluoromethyl)amine.[1]

Fluorinated amidoximes of the type $R_fC(NH_2)=NOH$ react with fluorinated acid chlorides in ether solution to give solid compounds of the general formula $R_fC(NH_2)=NOC(O)R_f$.[5] Fawcett[6] has reported that carbonyl fluoride reacts with bis(trifluoromethyl)amine in the presence of CsF catalyst at 300° and with  $\overline{(CH_2)_5C(O)NH}$ to give $(CF_3)_2NC(O)F$ and  $\overline{(CH_2)_5C(O)N}$-$C(O)F$, respectively.

Our investigations have shown that N,N-bis(trifluoromethyl)hydroxylamine and either CsF or KF form an adduct which reacts with acid halides to give products of the general formula $(CF_3)_2NOC(O)X$. Compounds where X = F, Cl, CF_3, C_3F_7, and $ON(CF_3)_2$ have been prepared and characterized. The nature of the $(CF_3)_2NOH$–metal fluoride adduct has been examined.

Experimental Section

Reagents.—Cesium fluoride (99%), trifluoroacetyl chloride, and heptafluorobutyryl chloride were obtained from K & K Laboratories. Carbonyl fluoride was prepared by the room-temperature reaction of an equimolar mixture of carbon monoxide (Matheson Co.) and fluorine and was purified by fractional codistillation.[7] Carbonyl chloride was obtained from the Matheson Co. and anhydrous potassium fluoride (Allied Chemical) was used. N,N-Bis(trifluoromethyl)hydroxylamine was prepared by reaction of $(CF_3)_2NONO$[8] with aqueous HCl[1] in the presence of mercury and was purified by fractional codistillation.

General Methods.—All reactions were carried out in 100-ml Pyrex bulbs connected to a stopcock through a 19/38 ₸ Pyrex joint. Prior to reaction the finely divided metal fluoride was placed in the bulb and heated at 200° for 10 min under dynamic vacuum to remove any water. The metal fluoride–N,N-bis(trifluoromethyl)hydroxylamine adduct was formed simply by condensing the hydroxylamine onto the fluoride at −183°. When the contents had warmed to room temperature, the acid chloride or the carbonyl compound was added using standard vacuum techniques. The reaction occurred smoothly and usually within 0.5 hr. Infrared spectra were obtained using a Beckman IR5A infrared spectrophotometer. All spectra were recorded in the gas phase with a Pyrex cell of 25-mm path length and sodium chloride windows. A Varian Model 4311B high-resolution nuclear magnetic resonance spectrometer was used to determine the nmr spectra at 40 Mc. Trichlorofluoromethane was used as an internal standard. Mass spectra were recorded using a Consolidated Engineering Corp. Type 21-103 mass spectrometer. Molecular weights were determined by Regnault's method. Vapor pressure data were obtained by holding the com-

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pound in a liquid state at various temperatures and reading the pressure on a mercury manometer. Boiling points of compounds with a low vapor pressure at room temperature were determined by refluxing them under an atmosphere of nitrogen. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, N. Y. The compounds reported are white solids and clear, colorless liquids. Infrared and mass spectra data are given in Tables I and II, respectively.

TABLE I

INFRARED SPECTRA AT 5000–625 CM^{-1}

$(CF_3)_2$-$NOC(O)F$	$(CF_3)_2$-$NOC(O)Cl$	$((CF_3)_2$-$NO)_2CO$	$(CF_3)_2NOC$-$(O)CF_3$	$(CF_3)_2NOC$-$(O)C_3F_7$
1917 s	1855 s	1890 s	1868 s	1858 s
1325 vs	1317 vs	1317 vs	1317 vs	1354 s
1273 vs	1269 vs	1269 vs	1268 vs	1321 vs
1242 vs	1225 vs	1237 vs	1242 vs	1268 vs
1220 vs	1035 vs	1218 vs	1188 vs	1248 vs
1190 vs	975 s	1132 vs	1070 vs	1230 vs
1073 s	876 m	1096 m	1033 w	1138 vs
976 s	714 s	1038 s	976 s	1083 s
960 m	643 m	975 s	878 m	1058 s
758 m		777 w	804 w	1033 s
715 m		748 w	757 m	976 s
669 m		713 s	717 s	957 s
645 w		668 w	697 m	942 m
			656 w	923 s
				854 s
				794 w
				752 s
				738 m
				714 s
				698 s
				658 m

TABLE II

MASS SPECTRA

Mass number, species, and relative abundance

$(CF_3)_2NOCOF^a$			$(CF_3)_2NOCOCl^a$			$((CF_3)_2NO)_2CO^b$			$(CF_3)_2NOCOCF_3^a$			$(CF_3)_2NOCOC_3F_7^b$		
215	$(CF_3)_2NOCOF^+$	(3.8)	212	$C_2F_5NOCO^{35}Cl^+$	(0.86)	250	$C_4F_6CO_3N_2^+$	(1.5)	246	$C_2F_6NOCOCF_2^+$	(4.9)	251	$C_5F_7CO_2N^+$	(4)
196	$C_2F_6NOCO^+$	(5)	196	$C_2F_6NOCO^+$	(22)	196	$C_2F_6NOCO^+$	(84.7)	196	$C_2F_6NOCO^+$	(22)	197	$C_3F_7CO^+$	(13)
168	$C_2F_6NO^+$	(2)				168	$C_2F_6NO^+$	(7)				169	$C_3F_7^+$	(98)
152	$C_2F_6N^+$	(1.3)	152	$C_2F_6N^+$	(2.2)	152	$C_2F_6N^+$	(10.5)	158	$C_2F_4NOCO^+$	(3)	150	$C_3F_6^+$	(2)
149	$C_2F_5NO^+$	(5.7)							149	$C_2F_5NO^+$	(13.4)			
133	$C_2F_5N^+$	(7.1)	133	$C_2F_5N^+$	(21)				133	$C_2F_5N^+$	(9.1)			
									130	$C_2F_4NO^+$	(16.4)	131	$C_3F_5^+$	(1)
114	$C_2F_4N^+$	(16)	114	$C_2F_4N^+$	(54)				114	$C_2F_4N^+$	(23.4)	119	$C_2F_5^+$	(12.5)
108	CF_2NOCO^+	(72)	108	CF_2NOCO^+	(18)	108	CF_2NOCO^+	(25.3)	108	CF_2NOCO^+	(3.6)			
									97	CF_3CO^+	(61)	100	$C_2F_4^+$	(12)
99	CF_3NO^+	(2.6)	79	$CO_2{}^{35}Cl^+$	(4.5)				78	CF_2CO^+	(5.2)	81	$C_2F_3^+$	(3.5)
69	CF_3^+	(1000)	69	CF_3^+	(1000)	69	CF_3^+	(1000)	69	CF_3^+	(1000)	69	CF_3^+	(1000)
63	FCO_2^+	(3.4)	65	$CO^{37}Cl^+$	(45)									
47	FCO^+	(275)	63	$CO^{35}Cl^+$	(156)									

[a] Consolidated Engineering Corp. Model 21-103 mass spectrometer, ionization potential 70 v. [b] Consolidated Engineering Corp. Model 21-620 mass spectrometer, ionization potential 100 v.

Preparation of Carbonyl Bis(trifluoromethyl)nitroxide [$((CF_3)_2NO)_2CO$].—A 6.0-mmole sample of $(CF_3)_2NOH$ was condensed into an excess of CsF (1–2 g). After this mixture had warmed to room temperature, 1 mmole of COF_2 was condensed onto the solid, and this was allowed to contact the solid for 15 min at room temperature. At the end of this time, the volatile products were removed. Six more 1-mmole fractions of COF_2 were added in the same manner and fractional codistillation of the products gave 2.5 mmoles of $((CF_3)_2NO)_2CO$. The molecular weight by vapor density was 354 ± 4 (calcd 364). The boiling point at 695 mm was found to be 70–71°. The F^{19} nmr spectrum consists of a single peak at ϕ 68.8. This compound did not react with water in the vapor phase for 24 hr at room temperature.

Anal. Calcd for $((CF_3)_2NO)_2CO$: C, 16.48; F, 62.64. Found: C, 16.49; F, 61.92.

Preparation of Bis(trifluoromethyl)nitroxocarbonyl Fluoride [$(CF_3)_2NOC(O)F$].—A 1.0-mmole sample of $(CF_3)_2NOH$ was condensed onto an excess of KF (CsF) (~1 g). After this mixture reached room temperature, 1.5 mmoles of COF_2 was condensed onto the solid. The reaction was allowed to proceed for 40 min, and fractional codistillation of the products gave 0.60 mmole of $(CF_3)_2NOC(O)F$ and 0.11 mmole of $((CF_3)_2N)_2CO$. The molecular weight by vapor density was 213 (calcd 215). A boiling point of 15° was found by extrapolation of a plot of log P *vs.* $1/T$. The data are [T (°C), P (mm)]: −79, 2; −52, 31; −44, 40.5; −19, 201; 0, 418; and 8, 575. The F^{19} nmr spectrum consists of a singlet at ϕ +68.8 assigned to the CF_3 fluorines and a singlet at ϕ +28.3 assigned to the CF fluorine. The peak areas were in the ratio of 6:1, respectively. This compound reacts immediately with water vapor at room temperature to give $(CF_3)_2NOH$ and CO_2 as the only volatile products.

Anal. Calcd for $(CF_3)_2NOC(O)F$: C, 16.74; F, 61.86. Found: C, 16.67; F, 61.48.

Preparation of Bis(trifluoromethyl)nitroxocarbonyl Chloride [$(CF_3)_2NOC(O)Cl$].—This compound may be prepared by two methods, the second of which is recommended. (a) A 0.50-mmole sample of $(CF_3)_2NOH$ was added to a small amount of CsF (~4:1) in an attempt to preclude the presence of free CsF, and this mixture was allowed to remain at 24° for several hours. A 1-mmole sample of $COCl_2$ was then added to the reaction bulb and the reaction was allowed to proceed at 200° for 12 hr since no reaction occurred at ambient temperature. Separation of the products gave 0.12 mmole of $(CF_3)_2NOH$, 0.025 mmole of $(CF_3)_2NOCOCl$, unreacted $COCl_2$, and small amounts of ClCOF, CF_3NCO, and $(CF_3)_2NOCOF$.

(b) To a 100-ml reaction bulb containing an excess of anhydrous $AlCl_3$ (~2 g) was added 0.64 mmole of $(CF_3)_2NOCOF$ and 0.64 mmole of HCl. The reaction was allowed to proceed for 38 hr at 200°. Separation of the products gave 0.10 mmole of $(CF_3)_2NOCOF$, 0.44 mmole of $(CF_3)_2NOCOCl$, and traces of $COCl_2$ and $(CF_3)_2NOH$. With a 10% excess of HCl, the reaction time may be reduced to 14 hr and the yield is somewhat higher. The molecular weight by vapor density was 224 (calcd 231). Partial vapor pressure data are [T (°C), P (mm)]: −46, 5; −28, 24; −16, 53; 0, 129; 6, 174; and 13, 227.5. Reaction of this compound with water vapor at room temperature gives $(CF_3)_2NOH$ and CO_2 as volatile products.

Anal. Calcd for $(CF_3)_2NOC(O)Cl$: C, 15.55; F, 49.30; Cl, 15.36. Found: C, 15.85; F, 48.93; Cl, 15.03.

Preparation of Trifluoromethylcarbonyl Bis(trifluoromethyl)nitroxide [$(CF_3)_2NOC(O)CF_3$].—A 0.75-mmole sample of $(CF_3)_2NOH$ was condensed onto an excess of CsF. With this mixture at 24°, 1.26 mmoles of $CF_3C(O)Cl$ was added to the solid and was allowed to react for 1 hr. Fractional codistillation of the products gave 0.14 mmole of $CF_3C(O)F$ and 0.68 mmole of $(CF_3)_2NOC(O)CF_3$. The molecular weight by vapor density was 268 (calcd 265). A boiling point of 30° was found by extrapolation of a plot of log P *vs.* $1/T$. The data are [T (°C), P (mm)]: −39, 21; −26, 50; −17, 85; −3, 174; 18, 469; and 23, 565.

The F^{19} nmr spectrum consists of two singlets, one at ϕ 68.6 assigned to the $(CF_3)_2N$ fluorines and one at ϕ 73.9 assigned to the CCF_3 fluorines. The peak areas are in the ratio of 2:1, respectively. Reaction of this compound with water vapor, after 1 hr, gave $(CF_3)_2NOH$ and CF_3COOH as the only volatile products.

Anal. Calcd for $(CF_3)_2NOC(O)CF_3$: C, 18.11; F, 64.53. Found: C, 18.26; F, 64.28.

Preparation of Heptafluoropropylcarbonyl Bis(trifluoromethyl)-nitroxide [$(CF_3)_2NOC(O)C_3F_7$].—A 1.2-mmole sample of $C_3F_7C(O)Cl$ was added to the hydroxylamine–metal halide adduct, which had been prepared in a manner analogous to that in the previous reaction. After 3 hr at 24°, fractional codistillation gave 0.10 mmole of unreacted $(CF_3)_2NOH$, 0.42 mmole of $(CF_3)_2NOC(O)C_3F_7$, and other products. The molecular weight by vapor density was 359 (calcd 365). The boiling point at 691 mm was found to be 65–67°. The F^{19} nmr spectrum consists of a singlet at ϕ +68.2 assigned to $(CF_3)_2N$, a triplet at ϕ +81.3 assigned to CF_3, a quartet at ϕ +118.3 assigned to $C(O)CF_2$, and a singlet at ϕ +127.0 assigned to the CF_2 group. The coupling constant between the CF_3 and the $C(O)CF_2$ is 8 cps. No hydrolysis occurs upon contact with water for several hours at room temperature.

Anal. Calcd for $(CF_3)_2NOC(O)C_3F_7$: C, 19.72; F, 67.67. Found: C, 19.12; F, 67.12.

Properties of the $(CF_3)_2NOH$–Metal Fluoride Solid.—This white solid is formed when $(CF_3)_2NOH$ is allowed to contact either CsF or KF. At room temperature the CsF adduct has an equilibrium vapor pressure of 0.5–1 mm of $(CF_3)_2NOH$, while that of KF exhibits a vapor pressure of 4 mm. The CsF adduct has a solubility in acetonitrile of approximately 0.4 g/ml and the solution can be conveniently removed from unreacted CsF which has a much lower solubility in this solvent. When the CsF is saturated with $(CF_3)_2NOH$, the latter is taken up until the solid which is formed consists of $(CF_3)_2NOH$ and CsF in a molar ratio of 2.1:1. The solid melts at $\sim$70° with slow evolution of $(CF_3)_2NOH$.

The F^{19} nmr spectrum of the CsF adduct in acetonitrile shows a single band at ϕ 67.6, attributed to the CF_3 fluorines, while the spectrum of $(CF_3)_2NOH$ in CH_3CN shows a single band at ϕ 69.0. The proton nmr spectrum of the CsF adduct shows a single band at -12.7 ppm relative to CH_3CN while $(CF_3)_2NOH$ in CH_3CN shows a band at -5.48 ppm relative to CH_3CN.

The solid remaining after reaction has not been examined extensively. We have found, however, that the remaining solid will not react further with $(CF_3)_2NOH$.

Results and Discussion

In Table I, the infrared spectra of the carbonyl compounds are given and several general assignments can be made. The bands in the region of 1855–1917 cm^{-1} can be assigned to the carbonyl group, and the bands at 975–976 cm^{-1}, characteristic of $(CF_3)_2N$ compounds, are assigned to C–N stretch. Comparison with spectra of similar compounds, such as $(CF_3)_2NOCF_3$ and $(CF_3)_2NCF_3$, where the former shows an absorption at 1070 cm^{-1} while the latter has no bands in this region, indicates that the N–O stretch in the compounds reported can be assigned to the strong band in the 1033–1075-cm^{-1} region. Other workers[4,8,9] have made similar assignments of the N–O stretching modes. For the compound $(CF_3)_2NOC(O)C_3F_7$, three strong absorptions in this region preclude such a definite assignment. The bands at 713–717 cm^{-1} are due to CF_3 deformation, and while definite assignments in the C–F region cannot be made, it is clear that three major absorptions at 1317–1325, 1268–1273, and 1225–1248 cm^{-1} are characteristic of each of the five compounds.

The mass spectral cracking patterns show an interesting similarity. In each case, except for $(CF_3)_2NOC(O)C_3F_7$, the $C(O)X^+$ group, from $(CF_3)_2NOC(O)X$, is second only to the very strong CF_3^+ in abundance. This indicates that a primary process which takes place is $(CF_3)_2NOC(O)X + e^- \rightarrow (CF_3)_2NO\cdot + C(O)X^+ + 2e^-$, which infers that the O–C bond is comparatively easily ruptured. This is not unexpected since the bis(trifluoromethyl)nitroxide radical is very stable.[2]

The F^{19} nmr spectrum of each of the compounds consists of a singlet at a rather nonvarying value of ϕ 68.2–69, owing to the $(CF_3)_2N$ fluorines. Blackley[2] has listed the shifts of the $(CF_3)_2N$ fluorines in a number of other compounds and these all fall in the region of ϕ 66–74. The chemical shifts of the other types of fluorines are consistent with what is expected for such perfluoroalkyl groups. That essentially no coupling occurs between the 1,2 fluorine atoms of the C_3F_7 group in $(CF_3)_2NOC(O)C_3F_7$ is not unusual. Others have also found this to be the case in compounds containing the C_3F_7 moiety, *e.g.*, in C_3F_7OF.[10] This has been explained on the basis of through-space coupling between fluorine atoms in a free chain.[11,12]

All of the compounds reported are stable in glass at room temperature; however, $(CF_3)_2NOC(O)F$, $(CF_3)_2NOC(O)Cl$, and $(CF_3)_2NOC(O)CF_3$ are hydrolytically unstable. This coupled with the fact that $((CF_3)_2NO)_2C(O)$ and $(CF_3)_2NOC(O)C_3F_7$ are both stable toward water indicates that steric hindrance is an important factor in their relative reactivities. In the series of compounds, $R_fC(NH_2){=}NOC(O)R_f$,[5] it was found that where $R_f = C_2F_5$, C_3F_7, and C_7F_{15} the compounds were stable toward water, although where $R_f = CF_3$ the compound was extremely susceptible to hydrolysis. This same trend is observed with perfluoroketones.[13]

Formation of the $(CF_3)_2NOH$–metal fluoride adduct is essential to the reaction with the acid halides. We have found bis(trifluoromethyl)hydroxylamine to react either very slowly or not at all with the acid halides in the absence of CsF or KF. We have further noted that if the hydroxylamine is not consumed in adduct formation because of a deficiency of metal fluoride, the excess $(CF_3)_2NOH$ does not react with the added perfluoroacyl halide.

In both the proton and F^{19} nmr spectra of the adduct a downfield shift was found in comparison with the bands in free $(CF_3)_2NOH$. If the compound were ionic in nature, the F^{19} shift would be expected to be upfield compared to the shift in free $(CF_3)_2NOH$. While we have no direct evidence that the proton band found in the adduct was necessarily due to a proton still bound to the hydroxyl oxygen, it would be reasonable

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to expect that it is in light of the F^{19} shift. Thus the compound is probably formed as the result of association of the protons with the fluoride of the CsF.

The solid remaining after reaction of the adduct with an acyl fluoride has been found to be thermally stable. Severe etching took place when a sample of gas, formed by thermally decomposing this solid, was introduced into an infrared cell with NaCl windows. It is likely that this solid could be $CsF\cdot 2HF$[14] but a mixture of other CsF–HF adducts cannot be excluded.

However, on the basis of the observation that a maximum of 2 moles of $(CF_3)_2NOH$ combines with 1 mole of CsF, we are led to believe that the reactions reported occur in two steps

$$n(CF_3)_2NOH + CsF \rightleftarrows ((CF_3)_2NOH)_n\cdot CsF \quad (n \leq 2) \quad (1)$$

$$((CF_3)_2NOH)_n\cdot CsF + nR_fC(O)X \longrightarrow n(CF_3)_2NOC(O)R_f + CsF\cdot nHX \quad (2)$$

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CONTRIBUTION FROM THE ISTITUTO CHIMICO DELL'UNIVERSITÀ, CENTRO NAZIONALE DI CHIMICA DEI COMPOSTI DI COORDINAZIONE ED ELEMENTO-ORGANICI DEL C.N.R., FERRARA, ITALY, AND THE ISTITUTO CHIMICO "G. CIAMICIAN" DELL'UNIVERSITÀ, CENTRO NAZIONALE DI CHIMICA DELLE RADIAZIONI E DEI RADIOELEMENTI DEL C.N.R., SEZIONE III, BOLOGNA, ITALY

Photochemistry of Coordination Compounds. XVI. Hexabromoplatinate(IV) and Hexaiodoplatinate(IV) Ions[1]

BY VINCENZO BALZANI, FRANCESCA MANFRIN, AND LUCA MOGGI

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The photochemical behavior of $PtBr_6^{2-}$ was investigated with radiation of 313, 365, 433, and 530 mμ, corresponding to electron-transfer and d–d bands of the complex. Spectrophotometric and pH measurements were used to establish the nature and the kinetics of the photoreaction. The results show that both d–d and electron-transfer excitations cause a photoaquation reaction. The quantum yield was found to be about 0.4, independent of the wavelengths of irradiation. The mechanism of the photoreaction is discussed, and it is proposed that the primary photochemical act is the heterolytic fission of a Pt–Br bond. On the basis of the constant value of the quantum yield, it is also suggested that the photoreaction originates from the same chemically active electronic state, independent of the wavelength of irradiation. The photochemical behavior of PtI_6^{2-} was investigated by irradiating at 254, 313, 365, and 530 mμ, which correspond to electron-transfer bands. The study was made difficult by the rapid thermal decomposition of the complex; however, it was proved that, also in this case, electron-transfer excitations cause a photoaquation reaction.

The photosensitivity of aqueous solutions of the hexahalo complexes of Pt(IV) has long been known.[2-6] Recently, studies have been reported on the photoexchange PtX_6^{2-}–X^- reactions[7-15] and on the behavior of flashed solutions of $PtBr_6^{2-}$ and PtI_6^{2-},[16] but no systematic investigations about the photochemical reactions of these complexes have been done. We wish to report the results obtained by irradiating $PtBr_6^{2-}$ and PtI_6^{2-} with radiation corresponding to their various absorption bands, in order to elucidate the mechanism of their photoreactions.

Experimental Section

Preparation of Complexes.—K_2PtBr_6 was prepared following the method of Gutbier and Bauriedel.[17] K_2PtI_6 was obtained according to Datta.[18] The complexes were recrystallized from dilute solutions of HBr and KI, respectively. The electronic absorption spectra of the complexes were found to agree well with the literature reports.[19] We tried also to prepare $PtBr_4$ following the method of Meyer and Züblin;[20] however, elemental analysis of the products obtained from different preparations showed Br:Pt ratios slightly higher than 4 and water contents between 20 and 30%.[21,22]

Apparatus.—Radiation of 254, 313, and 365 mμ was obtained using the irradiation equipment previously reported.[23] Narrow

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